

## IRS

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The following ASTM methods are used for the proximate analysis:

**ASTM D4239-94** Standard Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods

**ATSM D5373-93** (solids) Standard Test Methods for Instrumental Determination of Carbon, Hydrogen and Nitrogen in Laboratory Samples of Coal and Coke

**ATSM D5291-96** (liquids) Standard Test Methods for Instrumental Determination of Carbon, Hydrogen and Nitrogen in Petroleum Products and Lubricants

**ASTM D3179-89** Standard Test Method for Nitrogen in the Analysis Sample of Coal and Coke

**Water content** is determined by Loss on drying (LOD) for solids, Karl Fischer for liquids.

Results are reported on Dry Basis

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**ASTM D3174-93** Standard Test Method for Ash in the Analysis Sample of Coal and Coke

**ASTM D3175-89a** Standard Test Method for Volatile Matter in the Analysis Sample of Coal and Coke

**ASTM D5865-96** Standard Test Method for Gross Calorific Value of Coal and Coke

**ASTM D3302-02** Standard Test Method for Total Moisture in Coal

**ASTM D3172-89** Standard Practices for Proximate Analysis of Coal and Coke

### **ONLY for SOLID samples**

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#### **TGA Analysis**

Analysis is performed on one gram of sample. For the first raw coal sample, six different runs of one gram each are performed. For all synfuel samples and for any subsequent raw coal samples, only one run of one gram is performed. All the samples are dried in a desiccator overnight before performing the analysis.

The temperature program is as follows:  
30 °C/min from room temperature to 300 °C  
15 °C/min from 300 to 700 °C

The analysis should be run under a nitrogen atmosphere.

Sample size:

**Raw sample-** Approximately 8 grams of desiccated sample ground with wiggle bug (depending on the coarseness of the coal- WIGL 40 sec wait 40 sec and repeat) Put a portion of this material back in desiccator for FTIR analysis.

**Synfuel sample-** send ca. 2 grams of desiccated sample ground with wiggle bug (same process as raw).

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Note: The ASTM procedures cited in this document are followed by a notation outlining the year in which the specific standard(s) were revised (i.e. '-99') by ASTM. This is a common way to cite ASTM procedures and if the year is not current (i.e. '-03'), it does not indicate the procedure is 'out-of-date' since ASTM are not revised on a yearly basis. In some cases, the procedures outlined above are the most recent versions. In the remaining cases, the more recent version(s) are equally valid and substantive differences do not exist.

### **FTIR Analysis**

#### **ASTM E168-99** Standard Practices for General Techniques of Infrared Quantitative Analysis

- 1) As outlined in ASTM E168 A4, the sample(s) should be prepared using an internal standard.
- 2) The internal standard, sample, and KBR are pressed into a suitable pellet for analysis using transmission mode FT-IR.
- 3) The raw spectra are collected, labeled, and saved in an unmodified version for archiving purposes.
- 4) For ease of interpretation, the baseline is corrected using the methods outlined in section 12 of ASTM E168.
- 5) Peaks representative of the coal should be determined using the guidance set forth in ASTM E 168 7.1.7.
- 6) Peaks should be integrated for total area using the 2-point method, when possible.
- 7) To determine if a difference in the spectra of a coal and synfuel sample exist, the repeatability (i.e. std deviation) must first be determined. This may be different for different bands, so the %RSD should be

determined for every band utilized. This is typically determined using replicate analyses of the same sample and the %RSD calculated for each peak selected.

- 8) Comparison of samples (i.e. coal vs. synfuel) can then be accomplished by determining if the samples are different. Typically differences greater than 3 standard deviations are statistically significant (this is not the same significant, however).